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#### **MEMORANDUM**

DATE:

22 December 1998

TO:

David Bennett, WAM, U.S EPA, Region X

FROM:

Michelle Turner, Chemist, WESTON, Seattle

Roger McGinnis, Senior Environmental Chemist, WESTON, Seattle

SUBJECT:

Validation of Chlorinated Pesticide Data

Laboratory Batch K9805833

Site: Duwamish River

WORK ASSIGNMENT NO 46-23-0JZZ

WORK ORDER NO.:

4000-019-038-5200-00

DOC CONTROL NO.

4000-019-038-AAAK

cc.

Bruce Woods, RAP-WAM, U.S. EPA, Region X

Dena Hughes, Site Manager, WESTON, Seattle (memo only)

Kevin Mundell-Jackson, Database Management, WESTON, Seattle

The quality assurance review of three sediment samples, laboratory batch K9805833, collected from the Duwamish River has been completed. The samples were analyzed for chlorinated pesticides by Columbia Analytical Services of Kelso, Washington using EPA Method 8081. The samples were numbered:

98354045

98354049

98354052

#### **Data Qualifications**

The following comments refer to the laboratory performance in meeting the quality control criteria described in the technical specifications of the laboratory subcontract. The review follows the format described in the *National Functional Guidelines for Organic Data Review* (EPA OSWER Directive 9240 1-05, February 1994).

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QA Review Batch K9805833 (Chlorinated Pesticides) Site Duwamish River Page 2

#### 1 Timeliness

All samples met holding time criteria of 14 days for sample extraction and 40 additional days for extract analysis

## 2 GC/ECD Instrument Performance

i) Retention Time Windows

Retention times of all pesticides were within windows calculated from the initial calibration

ii) DDT/Endrın Breakdown

The percent breakdown for 4,4'-DDT and Endrin was less than 20 percent for each compound and combined breakdown was less than 30 percent on both GC columns.

#### 3 Initial Calibration

a) Individual Standard Mixtures

Retention time windows were calculated correctly.

Appropriate standards concentrations were used and peak heights of 50 to 100 percent of full scale were obtained.

Calibration factor percent relative standard deviation (%RSD) met QC criteria of 20 percent for pesticides and 30 percent for surrogates

#### 4 Calibration Verification

Instrument blanks and PEM samples were analyzed at the proper frequency

The difference between actual and calculated concentrations of individual pesticides was within QC criteria of  $\pm 25$  percent.

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#### 5 Detection Limits

Instrument detection limits met project required quantitation limits with the following exceptions:

Sample	Compound	QL Goal (µg/Kg)	Reported QL (µg/Kg)
98354046	Toxaphene	10	20

Where quantitation limit goals were exceeded, undetected analytes were qualified (UI) to indicate matrix interference.

## 6 Blanks

a) Laboratory Method Blanks

Laboratory method blank frequency criteria were met.

No target analytes were reported in laboratory method blanks.

b) Field Blanks

No field blanks were associated with this laboratory batch.

7. System Monitoring Compounds (Surrogates)

Surrogate compound percent recovery met quality control criteria for all samples.

8 Matrix Spike and Matrix Spike Duplicate

Matrix spike (MS) or matrix spike duplicate (MSD) percent recovery for the following compounds were outside QC guidelines (P-project, L-laboratory)

Sample	Compound	Percent Recovery	QC Limits
K9805842-008MS	gamma-BHC (Lindane)	38	46-127 (P)

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Site: Duwamish River

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Sample	Compound	Percent Recovery	QC Limits
(Batch QC)			28-123 (L)
K9805842-008MS (Batch QC)	Endrin	30	42-139 (P) 39-130 (L)
K9805842-008DMS (Batch QC)	gamma-BHC (Lindane)	43	46-127 (P) 28-123 (L)
K9805842-008DMS (Batch QC)	Endrin	30	42-139 (P) 39-130 (L)

Relative percent differences (RPD) between the MS and MSD percent recoveries were within QC guidelines for all spiked compounds No action was based solely on MS/MSD data.

# 9 Laboratory Control Sample (LCS)

LCS percent recoveries were outside the QC limits (P-project, L-laboratory) for the following compounds:

Sample	Compound	Percent Recovery	QC Limits
K980909-LCS	gamma-BHC (Lindane)	55	70-130 (P) 40-124 (L)
K980909-LCS	Heptachlor	54	70-130 (P) 40-117 (L)
K980909-LCS	Aldrın	54	70-130 (P) 43-108 (L)
K980909-LCS	Dieldrin	57	70-130 (P) 42-127 (L)
K980909-LCS	Endrin	55	70-130 (P) 46-123 (L)
K980909-LCS	DDT	64	70-130 (P) 46-127 (L)

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Results for compounds listed above were qualified as estimated (J). Undetected compounds were also qualified as estimated (UJ)

# 10. Field Duplicate Analysis

No field duplicate samples were associated with this sample delivery group.

#### 11 Second Column Confirmation

The relative percent difference (RPD) in reported analyte concentration was greater than 35 percent for the primary and confirmation column for the following samples:

Sample Number	Compound	DB-5 Conc (µg/Kg)	DB-1701 Conc (µg/Kg)	RPD
98354046	gamma-Chlordane	1 57	ND	NA
98354046	Endosulfan I	1 51	ND	NA
98354046	DDD	5 02	ND	NA
98354046	DDT	ND	3.05	NA
98354046	Methoxychlor	2 36	ND	NA
98354049	DDD	4 65	ND	NA
98354049	Methoxychlor	1 75	ND	NA
98354052	Methoxychlor	1 06	ND	NA

Differences can arise from analytical interferences on one column. However, the relative percent differences are not deemed significant at the reported concentrations. The lower concentration was reported for each analyte.

# 12. Sample Analysis

A cursory review of raw data was performed. All laboratory deliverables were present and complete. A duplicate analysis of Batch QC sample K9805842-008 was analyzed, all RPDs were less than 35 percent. The case narrative indicated that the MS and DMS recoveries of Endrin for the Batch QC was outside the laboratory QC limits due to

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probable matrix interference. As the LCS was within laboratory QC limits, no action was taken. No other problems were noted in the case narrative

13. Laboratory Contact

No laboratory contact was required

## Data Assessment

Upon consideration of the data qualifications noted above, the data are ACCEPTABLE for use except where flagged with data qualifiers that modify the usefulness of the individual values

#### Data Qualifiers

- U The compound was analyzed for, but was not detected
- UJ The compound was analyzed for, but was not detected The associated quantitation limit is an estimate because quality control criteria were not met.
- The analyte was positively identified, but the associated numerical value is an estimated quantity because quality control criteria were not met or because concentrations reported are less then CRDL or lowest calibration standard.
- R Quality control indicates that data are unusable (compound may or may not be present)
  Resampling and reanalysis are necessary for verification
- N Presumptive evidence of presence of material (tentative identification)
- I Elevated reporting limit due to matrix interference

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## COLUMBIA ANALYTICAL SERVICES, INC.

# Analytical Report

Client:

Roy F Weston, Inc

Project:

Duwamish River/4000-027-001-2019-38

Sample Matrix:

Sediment

Service Request: K9805833

Date Collected: 8/26/98 Date Received: 8/27/98

# Organochlorine Pesticides

Sample Name Lab Code

98354046 K9805833-007

Test Notes

Units ug/Kg (ppb)

Basis Dry

	Prep	Analysis		Dilution	Date	Date		Result
Analyte	Method	Method	MRL	Factor	Extracted	Analyzed	Result	Notes
alpha-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
beta-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-BHC (Lindane)	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND UJ	
Heptachlor	EPA 3550A	8081A	ī	1	9/9/98	10/2/98	ND ]	
Aldrin	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND 🗸	
Heptachlor Epoxide	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-Chlordane	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Endosulfan I	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
alpha-Chlordane	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Dieldrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND LUJ	
4,4'-DDE	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Endrin	EPA 3550A	8081A	2	I	9/9/98	10/2/98	ND WJ	
Endosulfan II	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDD	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Endrin Aldehyde	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Endosulfan Sulfate	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDT	EPA 3550A	8081A	2	1	9/9/98	10/2/98	NDUJ	
Endrin Ketone	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Methoxychlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Toxaphene	EPA 3550A	8081A	20	1	9/9/98	10/2/98	ND WI	ß

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The MRL is elevated because of matrix interferences

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Approved By

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Date 10/13/98

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# COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

Client:

Roy F Weston, Inc

Project:

Duwamish River/4000-027-001-2019-38

Sample Matrix:

Sediment

Service Request: K9805833

Date Collected: 8/26/98 Date Received: 8/27/98

# Organochlorine Pesticides

Sample Name Lab Code

98354049

K9805833-010

Units ug/Kg (ppb) Basis Dry

**Test Notes** 

	Prep	<b>Analysis</b>		Dilution	Date	Date		Result
Analyte	Method	Method	MRL	Factor	Extracted	Analyzed	Result	Notes
alpha-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
beta-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-BHC (Lindane)	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND UJ	
Heptachlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Aldrın	EPA 3550A	8081A	1	1	9/9/98	10/2/98	$\sqrt{QN}$	
Heptachlor Epoxide	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-Chiordane	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Endosulfan I	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
alpha-Chlordane	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Dieldrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND UJ	
4,4'-DDE	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Endrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND UJ	
Endosulfan II	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDD	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Endrın Aldehyde	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Endosulfan Sulfate	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDT	EPA 3550A	8081A	2	1	9/9/98	10/2/98	KJ (DN	
Endrın Ketone	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Methoxychlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Toxaphene	EPA 3550A	8081A	10	1	9/9/98	10/2/98	ND	

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Date 10/13/96

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## COLUMBIA ANALYTICAL SERVICES, INC.

## Analytical Report

Client:

Roy F Weston, Inc

Project:

Duwamish River/4000-027-001-2019-38

Sample Matrix:

Sediment

Service Request: K9805833

Date Collected: 8/26/98 Date Received: 8/27/98

# Organochlorine Pesticides

Sample Name

98354052

Lab Code Test Notes K9805833-013

Units ug/Kg (ppb)

Basis Dry

Analyte	Prep Method	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	Result Notes
						-		
alpha-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
beta-BHC	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-BHC (Lindane)	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND WJ	
Heptachlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Aldrın	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Heptachlor Epoxide	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
gamma-Chlordane	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Endosulfan I	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
alpha-Chlordane	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Dieldrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND UJ	
4,4'-DDE	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Endrin	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND UJ	
Endosulfan II	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDD	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Endrin Aldehyde	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Endosulfan Sulfate	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
4,4'-DDT	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND W	
Endrin Ketone	EPA 3550A	8081A	2	1	9/9/98	10/2/98	ND	
Methoxychlor	EPA 3550A	8081A	1	1	9/9/98	10/2/98	ND	
Toxaphene	EPA 3550A	8081A	10	1	9/9/98	10/2/98	ND	

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